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14. ABSTRACT

Methods for fabricating adaptive composite materials and components using shape memory alloy (SMA) constituents were investigated using a variety of fabrication techniques, including mechanical rolling methods. Both SMA-polymer and SMA-metal composites were created, as well as a new fabrication strategies for producing NiTi and CuAlNi shape memory alloy particles with refined size which still display shape memory and pseusoelasticty. The composites materials show promising facture toughness behavior. The functionalization strategy we developed for improving adhesion between a shape memory alloy constituent in a polymeric matrix provides tunable control over the interface. Additionally, related work on the manipulation and control of the motion of individual liberated nanowires using nickel end caps and magnetic fields has allowed us to achieve sample positioning for characterization and optical switching behavior.

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Final Performance Report

Nanostructured Shape Memory Alloys: Adaptive Composite Materials and Components

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Abstract

Methods for fabricating adaptive composite materials and components using shape memory alloy (SMA) constituents were investigated using a variety of fabrication techniques, including mechanical rolling methods. Both SMA-polymer and SMA-metal composites were created, as well as a new fabrication strategies for producing NiTi and CuAlNi shape memory alloy particles with refined size which still display shape memory and pseusoelasticty. The composites materials show promising facture toughness behavior. The functionalization strategy we developed for improving adhesion between a shape memory alloy constituent in a polymeric matrix provides tunable control over the interface. Additionally, related work on the manipulation and control of the motion of individual liberated nanowires using nickel end caps and magnetic fields has allowed us to achieve sample positioning for characterization and optical switching behavior.

Objectives

The objective of this research effort was to improve the application of shape memory alloys (SMAs) to microscopic structures and miniaturized devices by:

1) investigating the influence of grain refinement and cold working on the mechanical behavior of nanostructure SMAs produced by mechanical alloying;
2) exploring the use of the rolling and folding technique for the production of SMA nanparticles and SMA composite materials; and
3) optimizing the application of chemical functionalization for governing the adhesion of SMA particles in a composite matrix.

Composites involving shape-memory alloys (SMAs) afford the opportunity to investigate a variety of host-guest interactions at both molecular and bulk levels. We combined SMAs with various host matrix materials and using a variety of preparative and characterization techniques to determine the extent to which each component of the composite influences the other. Such material combinations have the potential to yield adaptive composite materials and components where the SMA elements either actively or passively control material and structural properties.

Accomplishments and New Findings

Discussed in detail in the sections below are the following recent developments:

- Developed a new mechanical rolling and folding fabrication strategy for producing NiTi and CuAlNi shape memory alloy particles
- Created and tested both polymer-SMA and metal-SMA composites
    - Fabricated composites using polypropylene, (PP), high density polyethylene (HDPE) and polycarbonate (PC) sheets as the matrix phase, and strips of NiTi or Ti as the inclusion.
    - Conducted pull out tests to characterize adhesion between NiTi or Ti and the polymer matrix materials being employed.
    - Established procedures to dissolve the polypropylene and the polycarbonate to separate the particles from the composites have.
    - Acquired preliminary results for damping, impact and fracture behavior.
    - Developed methods for incorporation of CuAlNi in a SnBi metal matrix.

A brief summary of other results obtained under this grant which have been published:

- Investigated transformation behavior in CuAlNi under nanoindentation conditions and correlated experimental findings with material deformation models to show that structures around indentation are austenite-martensite interfaces. Additionally a surface preparation technique was developed to provide minimal surface deformation necessary for observation of nanostructures.
    - W. C. Crone, H. Brock, A. Creuziger, "Nanoindentation and Microindentation of CuAlNi Shape Memory Alloy," *Experimental Mechanics,* 47, 133-142 (2007). Invited paper for special issue on nanomechanics.
- Developed a functionalization strategy for improving adhesion between a shape memory alloy constituent in a polymeric matrix, showing increased adhesion between SMA-polymer up to 400% and tunability of the SMA-polymer interface.
    - N.A. Smith, G. Antoun, A.B. Ellis, W.C. Crone, "Improved adhesion between a NiTi SMA wire and a polymer matrix via silane coupling agents," *Composites A*, Vol 35(11) 1307-1312 (2004).
    - G. Antoun, "Improving the Adhesion between NiTi Wire and Polymer Matrix for Composite Applications and Mechanical Fabrication of Nanometer-Sized Shape Memory Alloy NiTi Particles from Bulk by Rolling and Folding," M.S. Thesis, University of Wisconsin – Madison, 2003
    - Nick Smith, "Synthetic Approaches to Nanoscale Shape-Memory Alloys (SMAs) and Adhesion Properties of Composites Derived from Surface Modified SMAs," Ph.D. Thesis, University of Wisconsin – Madison, 2005
- Manipulated and controlled the motion of individual liberated nanowires using nickel end caps and magnetic fields, allowing for sample positioning for characterization, nanoscale device assembly, and optical switching applications.
    - A.K. Bentley, J.S. Trethewey, A.B. Ellis, W.C. Crone, "Magnetic Manipulation of Copper-Tin Nanowires Capped with Nickel Ends," *Nano Letters*, 4(3) 487-490 (2004).
    - A.K. Bentley, A. B. Ellis, G. C. Lisensky, W. C. Crone, "Suspensions of Nickel Nanowires as Magneto-Optical Switches," *Nanotechnology*, 16, 2193-2196 (2005).
    - A.K. Bentley, "Synthesis and Manipulation of Metallic Nanowires," Ph.D. Thesis, University of Wisconsin – Madison, 2005
- Reported the first observation of an interwoven austenite/martensite structure in a thin film TEM sample of NiTi. These observations are in contrast to the situation in bulk shape memory alloys where it is not possible to form an interface between austenite and a single variant of martensite.

    - L. Tan and W.C. Crone, "In situ TEM observation of two-stage martensitic transformation in aged NiTi shape memory alloy," Scripta Materialia, 50(6), pp.819-823 (2004).
- Identified a new strategy to maintain ultrafine grain sizes in multiphase microstructures.
    - N.A. Smith, N. Sekido, J.H. Perepezko, A.B. Ellis, W.C. Crone, "Synthesis of Dual Phase Bronze Alloys from Elemental Nanoparticle Constituents," *Scripta Materialia,* 51(5), 423-426 (2004).
- Produced improvements in wear behavior and increase in surface hardness for NiTi surface modified using oxygen and carbon ions
    - L. Tan, G. Shaw, K. Sriharan and W.C. Crone, "Effect of Oxygen Implantation on Wear Behavior of NiTi Shape Memory Alloy," *Mechanics of Materials*, 37, 1059-1068 (2005).

Mechanical Fabrication of SMA Particles

This research was initiated to address the scarcity of fabrication techniques for micro- and nano-sized NiTi shape memory alloy particles in an effort to produce shape memory particles for characterization and composite fabrication purposes. We used a method previously developed by co-PI Dr. John Perepezko for more conventional metals, to produce NiTi shape memory particles. In prior work we have also used the rolling and folding technique on nickel and titanium elemental foils to create metal-metal composites with shape memory function.[1] In the research supported by this grant, progressive cold rolling of thin shape memory alloy strips sandwiched between sheets of polymer was conducted to break up NiTi into a wide distribution of particle sizes. This method of creating particles can be applied with very little time investment, resources, or specialized equipment.

Different polymers were explored for the matrix phase: polypropylene (PP), high density polyethylene (HDPE), and polycarbonate (PC). The metal inclusion used was either a strips of nickel titanium alloy or titanium (used as the control). Each sandwich was prepared using two layers of polymer and placing a single metal or alloy strip between them. The sample was then subjected a single cycle of cold rolling in a rolling mill machine.

Very intriguing behavior is observed with this mechanical processing under the regimes investigated. Depending on the matrix/metal combination, the nip gap (space between rollers), and the rolling speed, the metal inclusion either displays no fracture, a wavy structure, or fracture as it is shown in Figure 1.

Observations of fracture in the NiTi and Ti strips show that the cracks propagate in direction of maximum normal stress. The multiple necking or multiple fracture of the harder composite constituent is believed to occur due to the instability that forms at the matrix/inclusion interface subjected to shear stresses.

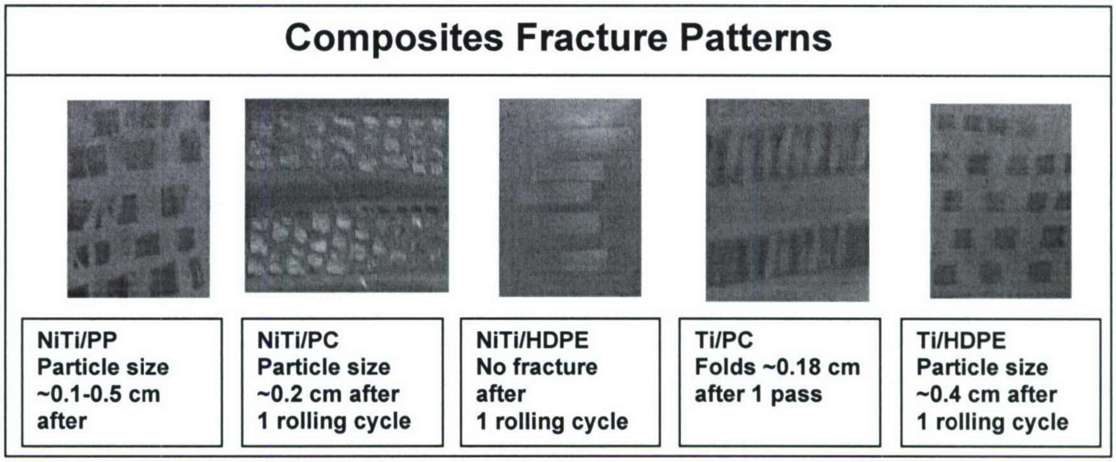

Figure 1: Metal inclusion fracture behavior.

Similar loading conditions for multilayer material have been reported in the literature for both composite materials and geologic materials.[2-5] There are overlaps in the theoretical approaches, but it is clear that numerous questions remain open. For instance, there is disagreement as to whether the relative moduli or the relative strengths of the polymer/metal couple is predictive of behavior.

In our research, we undertook an extensive experimental study of the effect of both material and processing parameters. Composite stress transfer theory, dimensionless analysis, and lubrication approximation theory (LAT) are being used to understand the fracture behavior of the layer-matrix systems under the cold rolling process (Figure 2). We have been investigating a dimensional analysis approach based on matrix transformation to identify important parameters. Figures 3 shows some of the results obtained in the parameterization study of the rolling conditions and matrix/inclusion material combinations being explored. In this case, a clear relation between strain and nip gap can be observed. The use of LAT on calendering of isothermal flow, a problem analogous to the experiments conducted, may allow us to predict regimes expected for the no fracture/instability/fracture behavior.[6-10]

This research is ongoing and will be a part of Barbara Calcagno's PhD thesis in Materials Science from the University of Wisconsin – Madison. She is currently supported on an NDSEG Fellowship.

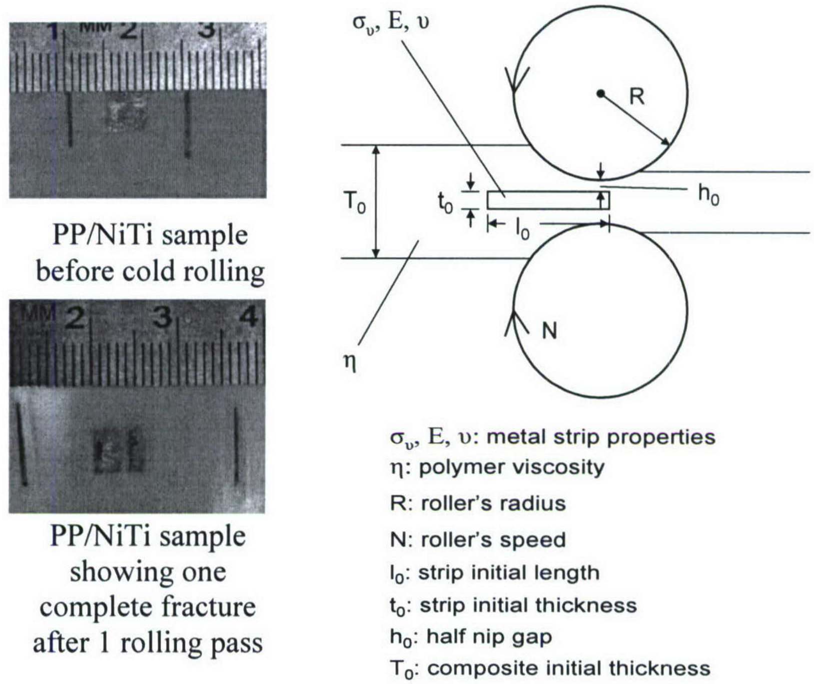

Figure 2: Cold rolling process.

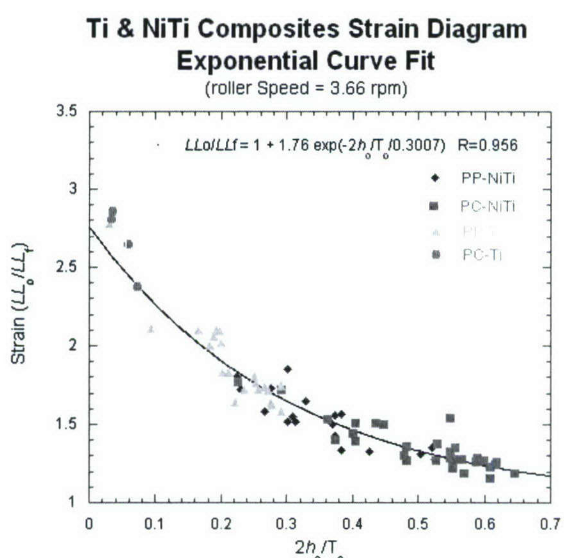

Figure 3: A plot showing the relationship between strain after rolling and the nip gap normalized with the initial sample thickness is shown for four different inclusion/matrix combinations at the rolling speed of 3.66 RPM.

Polymer-SMA Composites

Different composites were made using polypropylene (PP), high density polyethylene (HDPE), and polycarbonate (PC) as the matrix phase, and nickel titanium alloy or titanium as the inclusion phase. Each sandwich composite was prepared using two layers of the polymer sheet and placing the metal or alloy strips between them. The sample was then subjected to several cycles of cold rolling and folding to create a particle disperse composite by rolling the samples in a rolling mill machine. This process, shown schematically in Figure 4, involved placing the composites between two stainless steel sheets to minimize contamination. The process described was effective in breaking the nickel titanium and titanium strips in number of different matrix combinations with a range of process parameters.

In order to investigate the viscoelastic behavior of the composites, a Broadband Viscoelastic Spectrometer (BVS) was used to perform a preliminary analysis of the dynamic modulus and damping capacity behavior of a polymer containing NiTi particles and compared to the properties of homogenous polymer samples. A BVS is an inverted torsion pendulum capable to apply a torsion or bending load to a bar or cylinder that is fixed at one end. A small magnet is attached to the free end of the specimen and the load is applied via an oscillating magnetic field produced by running current through a Helmohltz coil centered around the magnet. A sample can be tested at different temperatures over a wide range of frequencies.[11] The dynamic tests were done with samples prepared with cold rolled PC and PP composites, no definite conclusions can be reported at this point.

In addition a sample of Methylmethacrylate and Styrene copolymer with embedded NiTi nanoparticles (0.05wt.%) as tested under torsion at 23°C, 44°C, and 60°C over a range of frequencies of 1-4kHz.. The viscoelastic response of this composite did not show any abnormal behavior under torsion loading as shown in Figure 5. However, it has been reported that a composite material with phase transforming barium titanate inclusions exhibited a viscoelastic modulus greater than that of either constituent in bending but not in torsion.[12] Further testing is required to determine if similar behavior is exhibited by composites with phase transforming NiTi inclusions.

In addition to viscoelastic testing, preliminary impact tests were performed on NiTi/PP and Ti/PP composites, and on PP samples (Figure 6). Results for mechanical properties such as Young's modulus, yield strength and ultimate strength for all composite constituents were also determined experimentally.

The adhesion strength between NiTi and several polymers were determined using wire pull-out tests. The polymers used were three different types of PP, two of PC and one of PS. The Melting Flow Index (MFI) parameter for all the polymers was measured following the ASTM standard.[13] Figure 7 shows the results obtained for the tests. For the NiTi samples the adhesion strength and the MFI of the polymers were plotted in Figure 8. The results show that the adhesion strength for the PP samples is only significantly affected under high MFI conditions (i.e. low molecular weight). Figure 9 provides a

comparison of the ratio between the ultimate strength of the inclusion material and adhesion between the inclusion and polymer matrix for several inclusion/matrix material combinations.

This research is ongoing and will be a part of Barbara Calcagno's PhD thesis in Materials Science from the University of Wisconsin – Madison. She is currently supported on an NDSEG Fellowship.

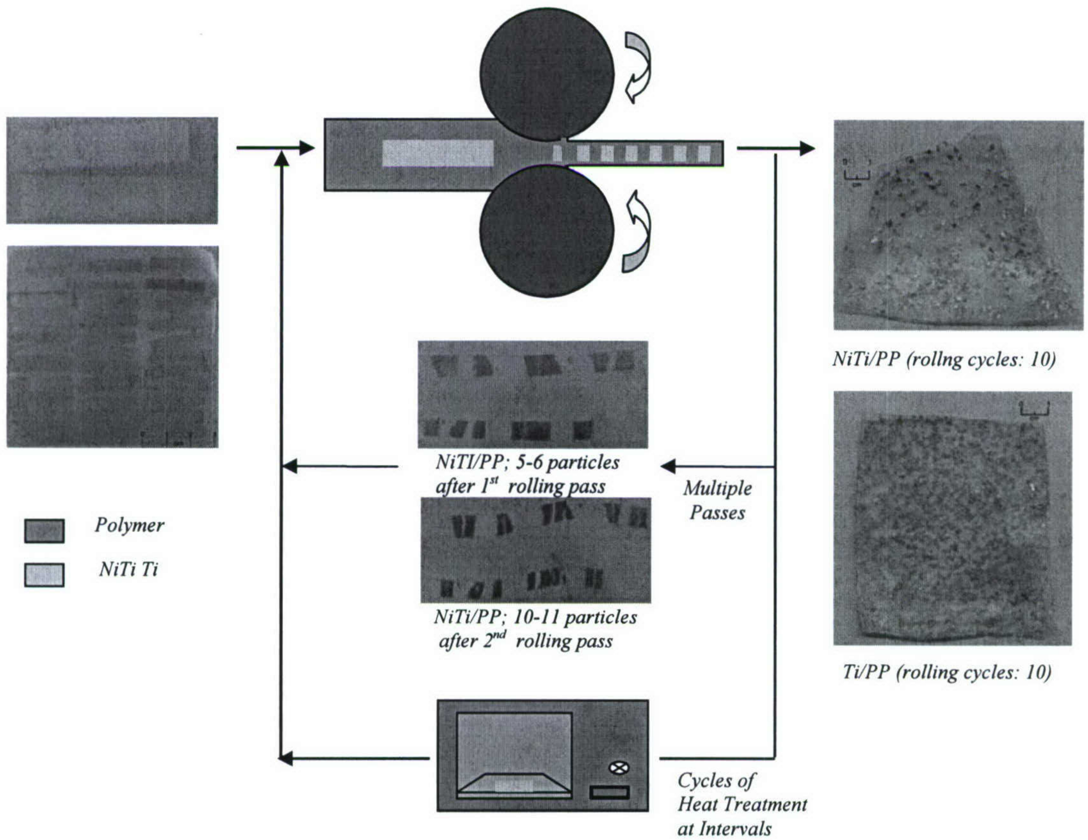

Figure 4: Processing procedure for polymer/metal composite fabrication.

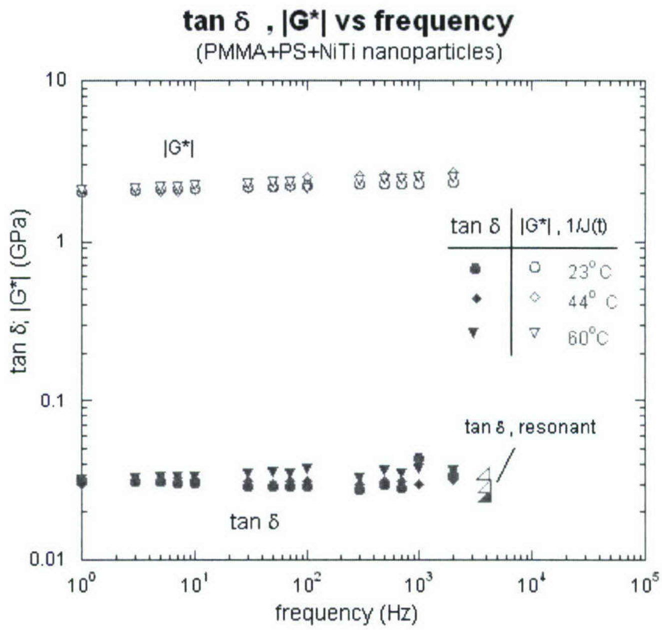

Figure 5: Dynamic properties (tanδ, |G*|) of NiTi polymer composite at 23°C, 44°C, and 60°C obtained with BVS

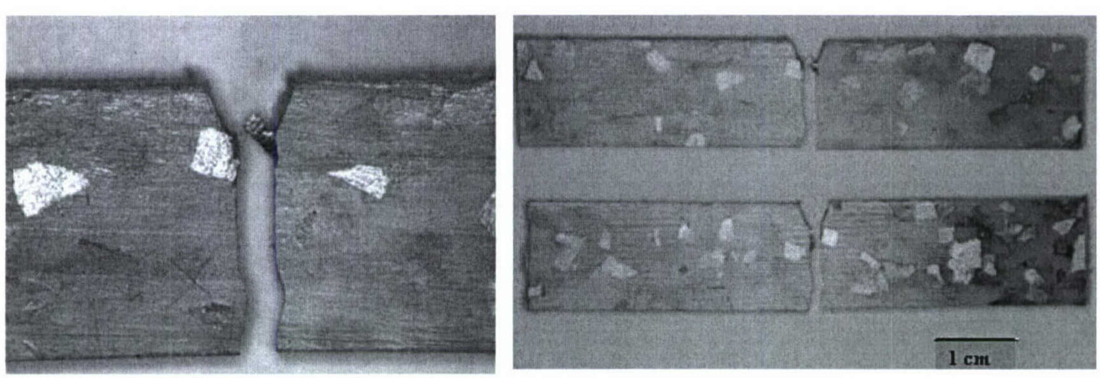

Figure 6: NiTi/PP samples after Izod impact test (10 rolling cycles)

Composite	Melting Flow Index (g/10 min)		Adhesion Strength (N/mm)	
(NiTi/polymer)	MFI	Std. Dev.	σ	Std. Dev.
PP-4.5 (pellets)	4.24	0.31	1.69	0.16
PP-20/30 (pellets)	18.44	0.91	2.7	0.3
PP- sheets	0.53	0.03	1.69	0.34
PC-pellets	12.1	0.71	8.9	1.5
PC-sheets	10.4	1.4	4.1	0.53
PS-pellets	1.5	0.2	5.1	0.5

Figure 7: Melting Flow Index and Adhesion Strength results for several NiTi/ polymer samples.

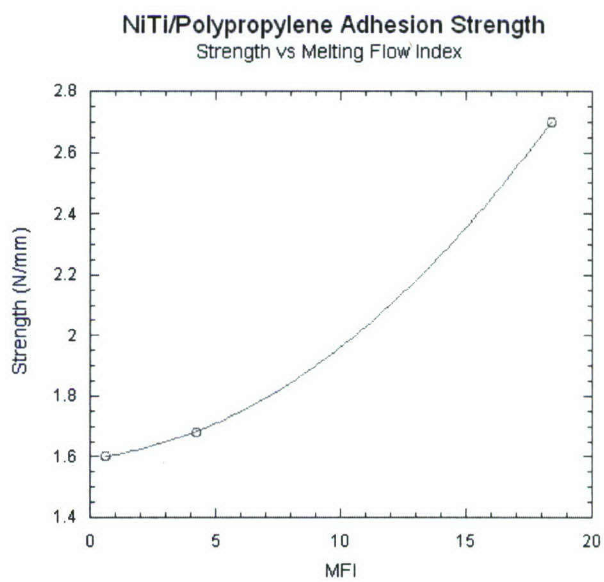

Figure 8: Adhesion strength and MFI for the PP samples.

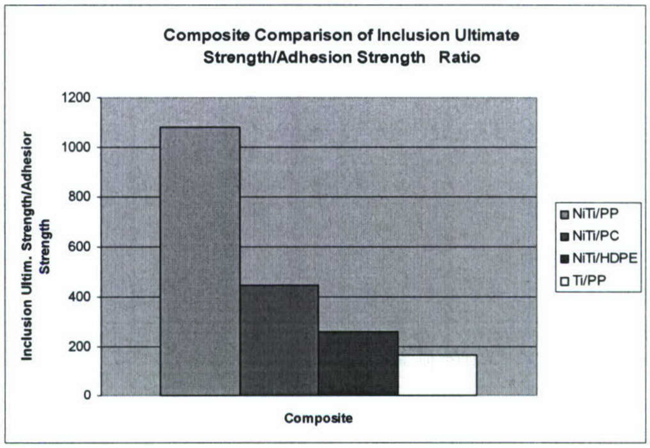

Figure 9. Comparison of the ratio between the ultimate strength of the inclusion material and adhesion between the inclusion and polymer matrix for several inclusion/matrix material combinations.

Metal-SMA Composites

A further mechanical fabrication method has been developed for creating CuAlNi (Cu82.12%-Al13.93%-Ni3.95%) shape memory particles on the millimeter to micrometer scale. Thin discs of CuAlNi 66.3mm in diameter were prepared by electrical discharge machining (EDM), with thickness of 1mm, which is on the order of the grain size. After cutting in to quarter pieced, the discs were heat-treated at 900°C for 1 hour and quenched in ice water. Each quarter piece was placed between two stainless steel sheets of 0.15 mm thick, which was processed with the rolling mill using a gap of 0.1 mm to fracture the material along the grain boundaries. This technique takes advantage of the poor intergranular fracture behavior of this material. After 10-15 rolling passes with the 0.1 mm gap dimension, particles with sizes between 220 and 380 μm were selected by mechanical sieving (Geotech Sandshaker). Part of the selected particles was sealed in quartz glass tube with vacuum inside, heat treated in furnace at 900 °C for 1 hour. This treatment imparted shape memory and pseudoelastic capability into the particles, which was verified by differential scanning calorimetry (DSC).

Prior to incorporation into the metal matrix, both heat treated and unheat treated particles were coated with pure Ni, which helped to improve wetability between the CuAlNi and BiSn. In the case of the composites, treated/untreated particles were added to a borosilicate glass vial. All subsequent processing condition for pure BiSn samples and the composite samples with treated/untreated particles were held constant. The BiSn was melted in the vial using a hot plate at 380 °C. Mechanical stirring at 5000 RPM for 2 minutes using a hand rotary tool with zigzag wire was used to induce mixing. After mixing, the samples were cooled by rinsing in tap water. Experiments varying particle

loading as well as control experiments using neat BiSn and BiSn with unheat treated CuAlNi particles were also conducted.

The rod-shape samples were removed by breaking glass vial, and cut with EDM to produce mechanical testing samples. The ASTM E399 disc-shaped compact specimen was created for fracture testing.[14] A chevron-shaped crack starter notch was created by fine wire (0.05 mm diameter) EDM.[15] One side of the notched sample was polished by Buehler® Minimet® Polisher, which provided good surface view for observation of microscopy. Diamond suspension (Buehler® Metadi®) of 9 micron, 3 micron, 1/4 micron were used.

A tensile load was applied by an Instron 5566 testing machine with a loading rate of 0.005 mm/s over a 2 mm travel range. The crack mouth opening distance was measured by a crack opening displacement (COD) gauges (Instron 2670-114). Load and displacement values were recorded automatically during the test. Crack propagation was observed by optical microscopy using a Nikon Eclipse at 5X, and digital images were taken every 5 seconds by a PixeLINK camera (PL-A662).

Initially, fracture testing was conducted on composites containing 0.64 wt% and 1.14 wt% particle loadings. The preliminary results shown in Figure 10 demonstrate a significant increase in fracture energy for samples containing the CuAlNi particles compared to the neat matrix. Following this initial study, a detailed study comparing the fracture energy for neat BiSn, BiSn with unheat treated CuAlNi particles, BiSn with heat treated CuAlNi particles and was conducted. The results shown in Figure 11 compare these three sample types (averages of a minimum of 3 samples tested under each condition and particle loading of 0.64 wt% for those samples containing particles). This data is plotted against crack mouth opening strain with is the preferred standard method. Figure 12 displays this same data as an area integration from the peak load, highlighting the differences in energy needed to propagate a crack under these conditions. Not only does the incorporation of particles induce a fracture toughening effect, the phase transforming capability of the heat treated particles substantially enhances the fracture toughness.

This research is ongoing and will be a part of William Lei Zhou's MS thesis in Engineering Mechanics from the University of Wisconsin – Madison.

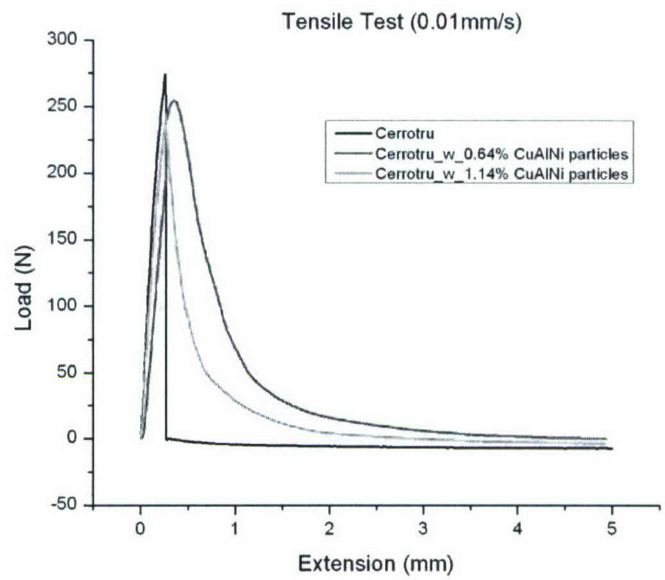

Figure 10: Load vs. crosshead extension curves for fracture tests of SnBi matrix containing various amounts of CuAlNi particles.

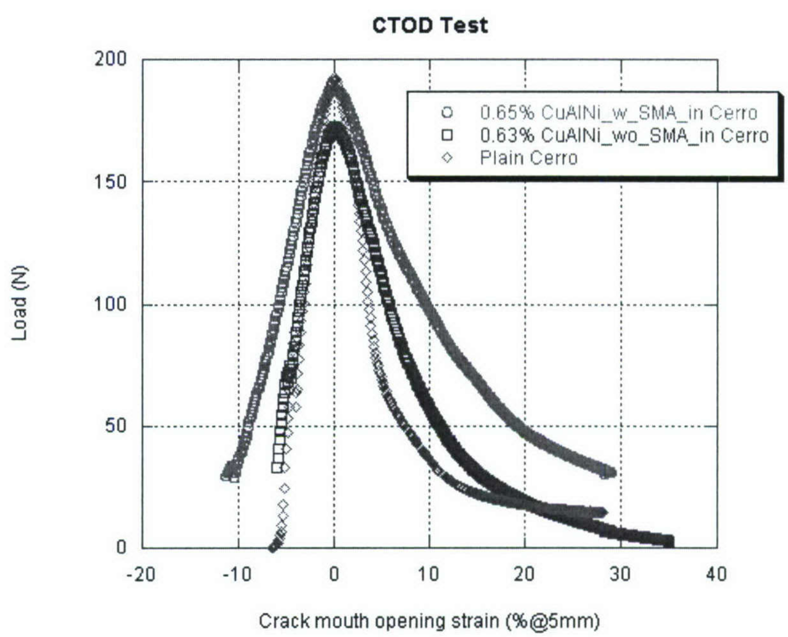

Figure 11. Load vs. crack mouth opening strain curves for fracture tests of three sample types: BiSn with heat treated CuAlNi particles, BiSn with unheat treated CuAlNi particles, and neat BiSn.

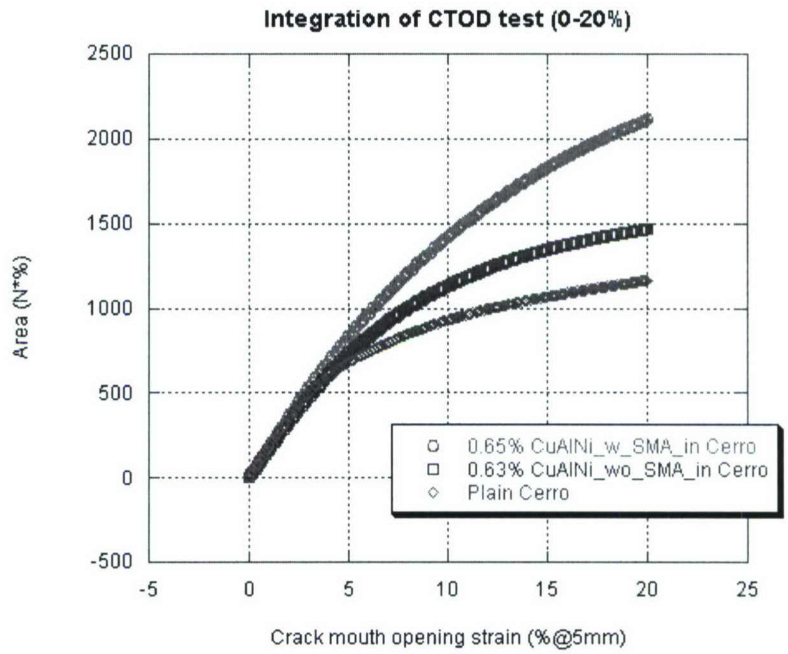

Figure 12. Integration of the area under the curve from the peak value of crack mouth opening displacement measured in fracture tests for three sample types: BiSn with heat treated CuAlNi particles, BiSn with unheat treated CuAlNi particles, and neat BiSn,

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Journal Publications

W. C. Crone, H. Brock, A. Creuziger, "Nanoindentation and Microindentation of CuAlNi Shape Memory Alloy," *Experimental Mechanics,* 47, 133-142 (2007). Invited paper for special issue on nanomechanics.

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A.K. Bentley, A.B. Ellis, and W.C. Crone (2005) Magnetic Manipulations of Nanowires, *Proceedings of the 2005 SEM Annual Conference and Exposition on Experimental and Applied Mechanics*, Portland, OR, 75:380, 1-4.

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L. Tan and W.C. Crone, "In situ TEM observation of two-stage martensitic transformation in aged NiTi shape memory alloy," *Scripta Materialia*, 50(6), pp.819-823 (2004).

Theses

Nick Smith, "Synthetic Approaches to Nanoscale Shape-Memory Alloys (SMAs) and Adhesion Properties of Composites Derived from Surface Modified SMAs," Ph.D. Thesis, University of Wisconsin – Madison, 2005

A.K. Bentley, "Synthesis and Manipulation of Metallic Nanowires," Ph.D. Thesis, University of Wisconsin – Madison, 2005

G. Antoun, "Improving the Adhesion between NiTi Wire and Polymer Matrix for Composite Applications and Mechanical Fabrication of Nanometer-Sized Shape

Memory Alloy NiTi Particles from Bulk by Rolling and Folding," M.S. Thesis, University of Wisconsin – Madison, 2003

Interactions and Transitions

a. Participation and presentations at meetings, conferences, and seminars

W.C. Crone, A. Creuziger, H. Brock, "Nanoindentation in CuAlNi Shape Memory Alloy," *2007 SEM Annual Conference and Exposition on Experimental and Applied Mechanics,* Springfield, MA.

W.C. Crone "Micro and Nanoscale Studies of Indentation in Shape Memory Alloys," Department of Mechanical Engineering, University of Wyoming, April 2007. Invited talk.

W.C. Crone, A. Creuziger, H. Brock, "Nanoindentation in CuAlNi Shape Memory Alloy," *Materials Research Society Spring Meeting,* San Francisco, CA.

W.C. Crone, "Nanoindentation in Shape Memory Alloys: Characterization and Application," Department of Mechanical Engineering, Johns Hopkins University, October 2006. Invited talk.

W.C. Crone, Symposium Co-Organizer: "International Symposium on MEMS and Nanotechnology," Society for Experimental Mechanics Annual Conference, June 2004, 2005, and 2006.

W.C. Crone, "Nanoindentation in Shape Memory Alloys: Characterization and Application," Department of Theoretical and Applied Mechanics Seminar, University of Illinois, Champaign/Urbana, February 2006. Invited talk.

W.C. Crone, "Nanoindentation in Shape Memory Alloys: Characterization and Application," ASM Regional Meeting, Wakesha, WI, April 2006. Invited talk.

W.C. Crone, Symposium Co-Organizer: "Mechanically Active Materials," Materials Research Society, Boston, MA, December 2004.

A.K. Bentley, A.B. Ellis, W.C. Crone, Magnetic Manipulations of Nanowires, 2005 SEM Annual Conference and Exposition on Experimental and Applied Mechanics, Portland, OR, June 2005.

A.K. Bentley, G.C. Lisensky, A.B. Ellis, W.C. Crone, "Nanoscience – Characterization Directed Light Scattering by Suspensions of Magnetic Nanowires," American Chemical Society, Spring 2005.

N.A. Smith, A.B. Ellis, W.C. Crone, "Nanoscale shape-memory alloys: Synthesis and applications" 228th American Chemical Society National Meeting, Philadelphia (2004).

W.C. Crone, L. Tan, "In situ TEM observation of two-step martensitic transformation in aged NiTi shape memory alloy," Society for Experimental Mechanics Annual Conference on Experimental Mechanics, Costa Mesa, CA, June 2004.

b. Consultative and advisory functions

The PIs are also involved with NSF-funded education efforts related to nanotechnology through the Materials Research Science and Engineering Center at the University of

Wisconsin – Madison and an Internships in Public Science Education grant. Research conducted under the AFOSR project has inspired several education projects. See http://www.mrsec.wisc.edu/edetc/, particularly laboratories located at http://www.mrsec.wisc.edu/Edetc/nanolab/nickel/index.html and http://www.mrsec.wisc.edu/Edetc/nanolab/jig/index.html.

c. Transitions

None at this time.

New Discoveries, Inventions, or Patent Disclosures

None at this time.

Honors and Awards Received by Key Personnel during the Grant Period

Wendy Crone was elected as a Fellow of the University of Wisconsin-Madison Teaching Academy, Fall 2006.

Barbara Calcagno was awarded National Defense Science and Engineering Graduate (NDSEG) Fellowship, selected by the Air Force Research Laboratory/Air Force Office of Scientific Research (AFRL/AFOSR) in 2006.

Henry Brock was awarded a National Security Education Program David L. Boren Graduate Fellowship in 2006 for study in Japan. He spent January through August of 2006 in the lab of Prof. Hideki Hosoda, Tokyo Institute of Technology, Japan.

John Perepzko was elected to the National Academy of Engineering, 2004.

John Perepzko was elected as Fellow of TMS, 2004.

Nick Smith received a Worldwide Universities Network Global Exchange Award to conduct research in Prof. Graham Leggett's laboratory at the University of Sheffield, Western Bank, Sheffield, S10 2TN, UK.

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